

## Conference Paper

# Gold Determination Problem in Barium Production Waste

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## Abstract

The most common method for gold content determination in raw materials is fire assay. An alternative to this method is atomic absorption in a solution obtained by dissolving an initial sample in *aqua regia*. This study focuses on the sludge of one of the chemical plants, in which the barite concentrate (barium in sulphate form) is fused with calcium chloride at coal presence, and then leached in water. Decomposition of samples in *aqua regia* followed by atomic absorption analysis, as well as fire assay, showed gold content in the sludge at the level of 1-4 gram per ton. The presence of infusible and chemically resistant compounds in the sludge does not allow to the reliable determination of the gold content. Sintering with sodium peroxide for decomposition of stable compounds at 700 ° C, dissolution of sinter in acidic solution, evaporation and fire assay of residue made it possible to establish a reliable concentration of gold in the sludge - 10-20 gram per ton. It was found that a direct determination of gold in acidic sinter leach solution provides inadequate values associated with the influence of the background.

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## 1. Introduction

Gold – is a noble metal, in human life it is of great importance. In the earth's crust is present exclusively in elemental form. In mineral ore raw materials can be in a free state, such gold is extracted using gravity methods. But more often, small particles of gold are interspersed in quartz or in sulfide minerals [1–4]. Such raw materials are processed by metallurgical methods.

In recent years, the so – called ultrafine gold, also mainly interspersed in the structure of the carrier mineral, has attracted special attention of specialists. Such a carrier can be quartz, sulfides, as well as organic matter, for example, coal, carbonaceous shales, peat. The size of finely disseminated gold does not exceed 10 microns. At fineness less than 0.1 µm gold is usually called nano–gold or colloidal gold [5, 6]. Known attempts to industrial extraction of such gold from coal and flue gases [7–10].

The physical properties of "nanogold" differ significantly from the usual gold. It is known that colloidal gold for a long time can be suspended in the liquid aqueous phase

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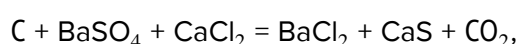
and in slag melts [11], passes through any filtering materials, and easily sublimates with increasing temperature [12]. The special properties of gold significantly complicate its extraction and do not allow to correctly assess the content of both the raw material and technological products.

A reliable assessment of the true gold content of minerals is an important and difficult task. It is known that several methods of analytical determination of gold in raw materials are used. The most common method is fire assay, which consists in melting a sample of ore or concentrate with fluxes (soda, borax, alkali) that provide liquid slag [13, 14]. Lead oxide and a reducing agent are introduced into the charge; at temperatures above 900-1000 °C, lead is reduced, which collects gold and settles to the bottom of the crucible. After separation of the noble metals from lead by weighing or spectral methods, the mass is determined and the content in the initial sample is calculated. This method allows to reliably determine how the fact of the presence of gold, and evaluate its content. However, if the composition of the charge is incorrectly selected and the conditions of the operations used are unsuccessful, the determination will be incomplete. There are numerous publications that testify to the loss of gold in the analysis with exhaust gases and slag during smelting [15].

An alternative to the gravimetric method, as the final stage of the fire assay analysis, is the atomic absorption determination of gold in a solution obtained by dissolving the initial sample or intermediate smelting product. Modern instruments make it possible to correctly determine gold at a concentration of less than 0.1 mg/dm<sup>3</sup>. The method is based on spraying the test sample with compressed air in a burner flame, while the ions of the metal being determined are atomized. The "atomic cloud" is illuminated by a beam of light from a special lamp that emits light of a certain wavelength. The absorption of this radiation by excited atoms, proportional to the concentration of the metal in an aliquot, registers the photomultiplier. Like fire assay, atomic absorption analysis may be accompanied by various kinds of systematic errors. If assay analysis is most often characterized by underdetermination, then atomic absorption can give a deviation both towards underdetermination and a result that is ten times higher than the true content. The reasons for obtaining overstated results are the subject of special studies.

## 2. Gold Determination

The object of the experiments in this work was the tailings of one of the chemical plants, in which the barite concentrate is fused with calcium chloride in the presence of coal:



The cake is leached in water, the  $\text{BaCl}_2$  solution is filtered off and processed to produce marketable products, and the undissolved residue is stored in dumps. The sludge consists mainly of two-water calcium sulfate, calcium hydroxide, unreacted  $\text{BaSO}_4$  and carbon. According to preliminary data, the sludge may contain gold introduced into the process by the initial components. Decomposition of samples in solutions of “aqua regia” and subsequent atomic absorption determination made it possible to obtain results from 1 to 4 g of gold per 1 ton of sludge. When using assay analysis in traditional modes, approximately the same indicators are recorded. Since the main components of the sludge are refractory and heat-resistant compounds  $\text{CaSO}_4$ ,  $\text{BaSO}_4$ , the production of liquid slag is difficult. This circumstance was the basis for doubts about the reliability of the results of preliminary analyzes.

It is assumed that the opening of samples of sludge in aqua regia does not provide a complete transfer of gold to the liquid phase. The reasons for the inaccessibility of gold to the effects of aqua regia are being studied. As an alternative, the efficiency of sintering of a sample with sodium peroxide, subsequent decomposition of cake in acidic solutions, and atomic absorption determination of gold in the final solution were tested [16]. In accordance with the methods recommended in this and other sources [8] for the analysis of such gold-containing raw materials, 1 g weighed samples were mixed with 1 g of sodium peroxide and sintered at a temperature of 700 °C for 1 hour. The resulting specs were cooled, crushed and leached with an acidic solution. The solutions after the necessary dilution were analyzed on an Analytik Jena novAA 300 spectrometer. Selected results of the analyzes obtained using the method used are shown in Table 1.

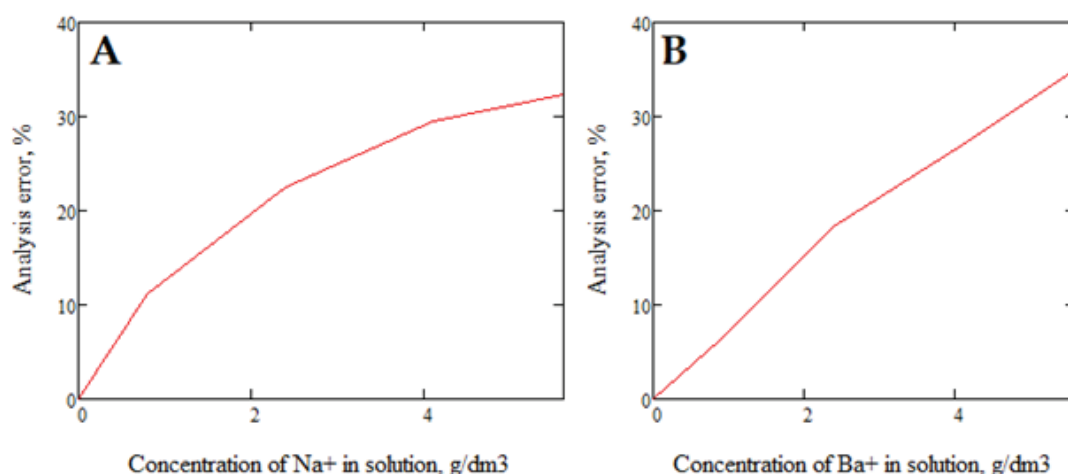
TABLE 1: Sludge analysis results by sintering

No	Sample weight, g	Solution volume, $\text{cm}^3$	Estimated Au content in the sludge, g/t
1	10	250	226.8
2	1	50	265.5
3	2	70	271.0
4	1	23	230.0
5	1	33	286.8

The estimated gold content is ten times higher than the results obtained by traditional methods. It is important that the data from parallel experiments strongly depended on the conditions and had a large discrepancy. At the same time, evaporation of the indicated solutions with a high gold content and fire assay analysis of the precipitates obtained in this way showed lower results than in the direct analysis of solutions, but significantly higher than the direct fire assay analysis of the initial slurry. These figures - from 10 to 20 g/t – can be regarded as close to the actual indicators. In this regard, the

problem of identifying the sources of error arises. Clarifying studies have shown that the reasons for obtaining pseudo-high results in the analysis of solutions of acid dissolution of cakes are several factors and, above all, the large background of  $\text{Na}^+$ ,  $\text{Ba}^{+2}$  and  $\text{Ca}^{+2}$  ions, the characteristic lines of the spectrum of which are superimposed on the gold spectrum, thereby introducing an error. In addition, at very high contents of these salts, crystallizing precipitates are deposited on the burner nozzle, which leads to a decrease in the flame intensity or distortion of its shape, and introduces additional errors. Additional dilution of the studied aliquots was not correlated with the obtained results. It was of interest to elucidate the quantitative effect of the background characteristics of alkali metals, primarily those that are present in solutions of the decomposition of cakes - Na and Ba on the results of atomic absorption gold. In these situations, it is recommended to use the additive method.

$\text{Na}^+$  cations in the form of NaCl salt were introduced to concentrations of 0.8; 2.4 and 4 g/dm<sup>3</sup> into a solution prepared from a standard gold sample with a gold content of 1.07 mg/dm<sup>3</sup>. If necessary, pH aliquots were adjusted with hydrochloric acid. The analysis data were compared with the true gold content and the error was calculated, the results are shown (Figure 1). In another series of experiments, the effect of  $\text{Ba}^{2+}$  ions were evaluated, and the  $\text{BaCl}_2$  reagent was used.



**Figure 1:** The effect of the concentration of  $\text{Na}^+$  and  $\text{Ba}^{2+}$  on the results of determination of gold in solution at its true content - 1.07 mg/dm<sup>3</sup> and a pH of 2.7

An analytical study of the solutions was carried out, the background of which simultaneously included sodium, barium, and calcium. The results obtained are ten times higher than the true gold content.

### 3. Conclusions

The true gold content in the slurry of chemical production significantly exceeds the indicators obtained using traditional methods. The reasons for obtaining high "apparent" indicators of atomic absorption determination of gold in solutions is the background of alkali metals. The greatest error in atomic absorption analysis of gold-containing solutions is introduced by barium.

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